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Modification of Sugarcane Bagasse Fiber into Microfibrils as Composite Materials Reinforcement

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Abstract. *Sugarcane bagasse, an abundant agricultural waste, holds great potential as a composite reinforcement material due to its sustainability and low cost. This study investigates the surface modification of bagasse fibers into microfibrils as reinforcement in polymer matrices. Alkalization using NaOH 7% (w/v) was applied to modify the surface of bagasse fibers. During the alkalization process, bagasse fibers were stirred with a magnetic stirrer at 200 rpm at 70° C for 5 hours to obtain micro-sized bagasse fibers (microfibrils). The alkalized bagasse fibers were morphologically tested using an electron microscope to determine their surface structure and diameter, then characterized using the Chesson method to determine their chemical composition. Finally, Fourier Transform Infrared Spectroscopy (FTIR) tests were performed to determine the functional groups of the bagasse fibers. The results showed that alkalization decreased bagasse fiber diameter, increased cellulose content, reduced the hemicellulose and lignin of the bagasse fibers, and reduced hydrophobic properties of bagasse fiber. Modified bagasse fiber has a diameter 0.35 μm, with a cellulose content 79.02%, hemicellulose 0.82%, and lignin 10.15%. The decrease in hydrophobic properties was shown by decreasing the number of hydroxyl functional groups (-OH). Based on the results obtained, modification of bagasse fiber using NaOH 7% (w/v) effectively produces microfibrils.*

Keywords: Alkalization, Bagasse fibers, Microfibrils.

Introduction

Natural fiber-reinforced composites have garnered significant attention recently due to their environmental benefits, cost-effectiveness, and wide availability. These materials serve as a sustainable alternative to synthetic composites, addressing global resource depletion and environmental degradation challenges. Among the various types of natural fibers, sugarcane bagasse fiber stands out due to its abundant availability as a byproduct of the sugar industry. Globally, millions of tons of sugarcane bagasse are produced annually, much of which remains underutilized or disposed of as waste, creating environmental concerns [1].

The utilization of sugarcane bagasse as a reinforcement material in composite applications presents a promising solution for waste management and sustainable material development. Sugarcane bagasse fibers possess advantageous properties such as low density, biodegradability, and a high cellulose content, making them suitable for improving the mechanical and thermal performance of composites. However, the direct use of raw sugarcane bagasse fibers often presents challenges, including inconsistent fiber properties and limited interfacial adhesion with polymer matrices [2], [3], [4].

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To overcome these limitations, reducing the size of sugarcane bagasse fibers to microfibrils is essential. Converting sugarcane bagasse fibers into microfibrils enhances their surface area, improves their mechanical properties, and facilitates better compatibility with composite matrices. Microfibrils derived from sugarcane bagasse exhibit uniform morphology and superior structural integrity, making them an ideal reinforcement material for high-performance composite applications [5].

The main factor that determines the success of fiber as reinforcement is the bond formed between the fiber and the matrix, the better the bond, the stronger the adhesion force of the reinforcement and the matrix so that the better the characteristics of the resulting composite material [6], [7], [8]. Bagasse fibers are one of the natural fibers that can improve mechanical properties such as tensile strength, flexural strength, flexural modulus, hardness, and impact strength of a material when appropriate fiber modifications are made [9]. The capability of bagasse fiber composites depends on several factors, including fiber chemical composition, structure, physical properties, mechanical properties, and fiber interaction with the polymer. The average tensile strength of bagasse fiber is 290 MPa, Young's modulus is 17 GPa, and the density is 1.25 g/cm^3 [10]. Bagasse fiber contains about 50% cellulose, 25% hemicellulose, and 25% lignin. Bagasse is a lignocellulosic material that provides an abundant and renewable source of energy [11].

Surface modification of sugarcane bagasse fibers has emerged as a pivotal area of research in developing high-performance natural fiber-reinforced composites. Alkali treatment, specifically using sodium hydroxide (NaOH), is a commonly employed method due to its effectiveness in enhancing fiber properties by removing impurities such as lignin, hemicellulose, and wax. This review highlights recent studies focusing on alkali-treated sugarcane bagasse fibers and identifies areas requiring further development to optimize their applications in composite materials [11].

Anggono [12], focuses on enhancing the mechanical properties of green composites reinforced with sugarcane bagasse fibers (SCBF) through alkali treatment using a 10% NaOH solution. The study specifically investigates the impact of varying alkali soaking times on the fiber's surface morphology, tensile properties, and fiber-resin bonding within a polypropylene (PP) matrix. The study involved the preparation of SCBF by soaking the fibers in 10% NaOH at 60–70°C for 2 to 6 hours. Treated and untreated fibers were mixed with PP in weight ratios of 20:80, 25:75, and 30:70. Hot pressing was employed to produce composite samples, which were then subjected to tensile testing. SEM analysis was conducted to examine changes in surface morphology and interfacial bonding between the fibers and the PP matrix. The alkali-treated SCBF composites exhibited up to a 43% improvement in tensile strength compared to untreated samples. The study only utilized SEM in the analysis of alkalized fibers. Incorporating advanced tools like XRD or FTIR could provide deeper insights into structural changes post-treatment [12].

Similar to previous research, Devadiga et al. [13] explore advancements in sugarcane bagasse fiber-reinforced composites, emphasizing bagasse as a sustainable reinforcement material in polymer matrices. It reviews chemical and alkali treatments to enhance fiber properties and optimize composite performance. The research examines various chemical treatments, such as alkali, silane, acetylation, and permanganate, to improve interfacial bonding, reduce hydrophilicity, and enhance the mechanical properties of bagasse fibers. Sodium hydroxide treatment significantly improves fiber surface roughness and reduces lignin and hemicellulose content, leading to enhanced fiber-matrix adhesion [13]. The research has not focused on the

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production of bagasse microfibrils, so further analysis of the surface modification of bagasse fibers is needed.

This study evaluates the effectiveness of alkali treatment using sodium hydroxide (NaOH) to produce sugarcane bagasse microfibrils with enhanced characteristics. The alkali treatment aims to increase the cellulose content of the fibers while significantly reducing hemicellulose and lignin levels. Additionally, the method seeks to enhance the hydrophobic properties of the fibers, which is a critical indicator of the effectiveness of the alkali treatment in improving fiber characteristics. By exploring these parameters, the study aims to provide a robust understanding of the potential of alkali-treated sugarcane bagasse microfibrils in composite material applications.

Theoretical Background

The thing that must be considered when making composite materials is the ability of the fiber to bond with the matrix (interfacial bonding). The stronger the interfacial bonding between the fiber and the matrix, the better the characteristics of the composite material. One way to improve interfacial bonding between fiber and matrix is to treat the fiber called fiber surface modification. The fiber surface can be chemically modified by various methods, in general, the methods that are often used are fiber modification using chemical, heat treatment, and enzymatic methods. Chemical fiber modification includes alkalization, acetylation, coupling agent, permanganate, and others [14]. In this study, the fiber modification used is alkalization treatment.

Alkalization Treatment

Alkalization treatment is one of the most widely used chemical treatments of natural fibers. Alkalization treatment can remove impurities on the fiber surface and make the fiber diameter smaller [15]. Alkalization treatment is not only able to improve mechanical properties, make fibers have a rough surface, and reduce the water absorption of composite materials but also to increase the amount of amorphous cellulose [16].

In addition to increasing the amount of amorphous cellulose, alkalization is also able to eliminate hydrogen bonds in the network structure thereby reducing the hydrophilic nature of the fiber [15]. The following reaction occurs in the alkalization process (1):

$$\text{Fibers} - OH + NaOH \rightarrow \text{fibers} - O - Na^{+} + H_2O \qquad (1)$$

The penetration of sodium hydroxide into the crystalline region of the parent cellulose results in alkalized cellulose. Then, after washing away the unreacted NaOH, the formation of regenerated cellulose occurs [17], [18], [19], [20].

Alkalization of natural fibers is a critical process that can yield microfibrillated and nanofibrillated fibers, as illustrated in the following schematic (**Figure 1**). This process involves treating raw natural fibers with a sodium hydroxide (NaOH) solution, effectively removing lignin, hemicellulose, and other non-cellulosic impurities. By eliminating these components, the internal structure of the fibers is disrupted, leading to the separation of microfibrils.

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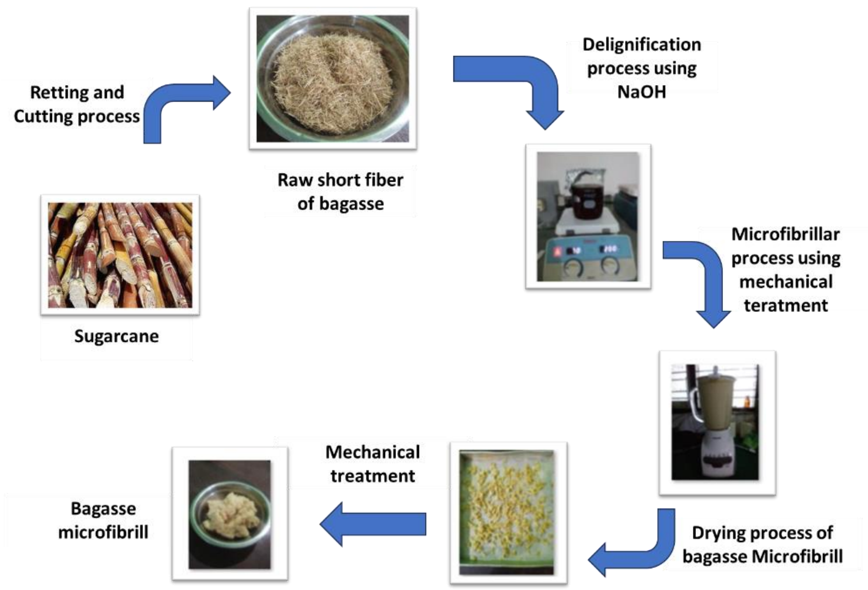

Figure 1. Schematic of the microfibrillar process.

Through further mechanical or chemical treatments, such as homogenization or enzymatic hydrolysis, these microfibrils can be broken down into finer structures, resulting in cellulose nanofibrils (CNFs). CNFs are characterized by their nanoscale dimensions, exceptional strength, and high surface area, making them valuable for a wide range of applications, including composites, biomedical materials, and nanotechnology [21], [22].

The alkalization process enhances the fibers' purity and functionality and improves their compatibility with other materials in composite formulations. This makes it a vital step in the development of sustainable materials derived from natural sources. The transformation from raw natural fibers to microfibrils and nanofibrils underscores the versatility and potential of cellulose-based resources in advancing green technologies [6].

Materials and Methods

The materials used in this research are bagasse fiber, NaOH, water, aluminum foil, distillate water, H_2SO_4, and acetic acid. The bagasse fibers used were obtained from the disposal waste of the Semboro Sugar factory, Jember, East Java. Utilizing waste from the Semboro Sugar factory demonstrates a specific effort to address local sustainability issues. This also provides an advantage in this study, where the materials obtained become cheaper.

In this study, the use of FTIR spectroscopy offers a more detailed understanding of chemical modifications, such as cellulose crystallinity and hemicellulose/lignin removal. This is absent in the previous study. The incorporation of acids (H_2SO_4 and acetic acid) for neutralization after NaOH treatment potentially provides a more balanced chemical profile of treated fibers compared to methods that rely solely on water rinsing, which was not done in the previous study.

The method used in this research is the alkalization process, using 7% w/v NaOH, applied to the bagasse fibers. The fibers were first washed with running water to remove any dust or impurities.

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Afterward, the fibers were blended to separate any cork still attached to the bagasse. The blended fibers were then dried under the sun and cut into 1-2 cm lengths before undergoing alkalization treatment.

The previous study utilized NaOH at a higher concentration (10% w/v), while this study used 7% (w/v) NaOH. Additionally, this study included a pre-treatment process for the bagasse fibers before alkalization, which was not performed in the previous study. A visualization of this alkalization method is shown in **Figure 2**.

Alkalization Treatment

Figure 2. Alkalization of bagasse fiber

The alkalization process was carried out using a 7% (w/v) NaOH solution, with a magnetic stirrer set at 200 rpm and heated to 70°C for 5 hours. The alkalized fibers were then blended for 5 minutes and thoroughly washed until neutral, with a few drops of acetic acid. After being sun-dried, the fibers were oven-baked at 100°C for two 15-minute intervals. The dried bagasse fibers were then mixed to separate them and prepared for characterization.

Morphological Characterization of Bagasse Fiber

This investigation used the Metkon IMM 902 electron microscope to examine the surface morphology of bagasse fiber. The test was carried out by placing bagasse fibers from the alkalization of NaOH on the microscope table. Next, the magnification was chosen; in this investigation, a 20x magnification was utilized to observe the bagasse fiber's structure, roughness, and diameter. The recording of the bagasse fiber observation results is represented in the form of a 2-dimensional image. The picture shows the effect of alkalization treatment with variations in NaOH concentration on the surface morphology of bagasse fibers such as structure roughness and diameter of bagasse fibers. The diameter of the bagasse fiber is determined by selecting several points on the fiber, the diameter value displayed in the image is then divided by the magnification of the microscope used, and then several diameters are averaged to determine the actual diameter of the bagasse fiber.

Chemical Composition Testing of Bagasse Fibers

The chemical composition of alkalized bagasse fiber was examined to determine the levels of cellulose, hemicellulose, and lignin degraded using the Cheson method.

a. To obtain the dry sample (a), 1-2 grams of material were added to 150 mL of distilled water, heated to 100°C for 2 hours, filtered through filter paper, and rinsed with water. The solid

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residue was then dried at 105°C and weighed. After one hour of heating at 100°C, the sample was treated with 150 mL of 1N H_2SO_4, filtered through filter paper, and washed with distilled water. Additionally, a dry sample (b) was obtained by weighing the solid portion after it was dried at 105°C. Equation 2 was then used to determine the hemicellulose content.

$$\text{Hemicellulose content} = \frac{\text{(a)-(b)}}{\text{initial weight}} \times 100\% \qquad (2)$$

b. Second, the amount of cellulose was measured by immersing the dry sample (b) in 10 ml of 72% H_2SO_4 for 4 hours, followed by the addition of 150 ml of 1N H_2SO_4, which was heated to 100°C for 2 hours, filtered through filter paper, rinsed with distilled water, and dried in an oven at 105°C to produce a dry sample (c). Equation 3 is then used to determine the cellulose content.

$$\text{Cellulose content} = \frac{\text{(b)-(c)}}{\text{initial weight}} \times 100\% \qquad (3)$$

c. Lastly, heat the dry sample (c) to 600°C for 4–6 hours, weigh it, and extract the ash (d) to ascertain the lignin content. Equation 4 then determines the lignin level.

$$\text{Lignin content} = \frac{\text{(c)-(d)}}{\text{initial weight}} \times 100\% \qquad (4)$$

Chemical Characterization of Bagasse Fiber (FTIR Analysis)

The function group of a substance is determined by FTIR (Fourier Transform Infra-Red) analysis based on the level of absorption created. When light is absorbed by a substance, it transfers its frequency to that compound. The chemical's molecular state will be altered by the amount of energy absorbed. Because each link demands a different quantity of energy, it absorbs infrared light at varying frequencies. The transmittance % will be used to assess the number of frequencies that are not absorbed by the substance [23]. In this study, bagasse fiber samples were analyzed using FTIR without alkalization at 0% NaOH and with alkaline treatment at 7% NaOH concentration. The purpose of this characterization is to investigate the effect of alkaline treatment on the functional groups present in bagasse fiber.

Results and Discussion

Surface morphology of bagasse fibers

The sugarcane fiber surface morphology (**Figure 3**) was observed using an electron microscope. Observations were made to determine the effect of alkalization on the structure and diameter of bagasse fibers. The bagasse fibers used in this observation are fibers before and after alkalization (using NaOH 7% (w/v)). The untreated fiber shows a smooth and intact surface with minimal disruption. The diameter measures approximately 3.7 µm, indicating the presence of natural non-cellulosic components, such as lignin, hemicellulose, and other impurities, which contribute to the

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structural integrity and bulkiness of the fiber. The surface appears uniform but lacks significant roughness, which may result in poor interfacial bonding when used as a reinforcement in composite materials.

(a) (b)

Figure 3. The diameter of Bagasse fiber (a) before alkalization 10× magnification is 3.7μm and (b) after alkalization 7% (w/vol) % NaOH 20× magnification is 0.35 μm.

Figure 3 shows changes in the structure and diameter of bagasse fibers after alkalization treatment, where the fiber surface structure becomes cleaner and the fiber diameter becomes smaller after alkalization treatment. The fiber's surface becomes cleaner indicating that impurities are removed during the alkalization process and alkalization treatment causes the fiber diameter to become smaller [6]. The results show that the diameter of bagasse fibers after alkalization is ± 0.35μm. In general, when the fiber is smaller, the strength of the composite material is higher [24]. Decreasing the smaller fiber's diameter increases the fiber's interfacial area in the composite material. The large interfacial area greatly influences determining the final characteristics of the composite material [25].

Chemical Composition of Bagasse Fibers

The Cheson method was employed in this study to evaluate the quantities of cellulose, hemicellulose, and lignin in bagasse fiber, allowing for an understanding of how alkaline chemical treatment affects the chemical composition of the fiber. This analysis is crucial because the chemical composition of the fiber influences its properties. According to Jose et al. [26], the mechanical characteristics of plant fibers are primarily determined by the cellulose content. This indicates that the mechanical properties of the fiber improve as its cellulose content increases. However, further testing is required, as various factors influence the properties of composite materials. Therefore, additional tests are necessary to determine the optimal fiber qualities that will yield superior composite material properties. Meanwhile, **Table 1** presents the results of the chemical composition analysis of bagasse fiber in this study.

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Table 1. Chemical composition of bagasse fiber after alkalization 7 % (w/vol) % NaOH

Sample	Bagasse fiber
Hemicellulose content (%)	0.82
Cellulose content (%)	79.02
Lignin content (%)	10.15

Based on **Table 1**, it is known that after 7% (w/vol) NaOH alkalization, bagasse fiber has a cellulose content of 79.02%, hemicellulose 0.82%, and lignin 10.15%. This shows better results when compared to the reference which is about 50% cellulose, 25% hemicellulose, and, 25% lignin [11]. Fiber chemical composition changes after alkalization treatment are caused by the decomposition of lignin, hemicellulose, and cellulose. As the fiber deteriorates, its chemical composition disintegrates, with lignocellulose reacting with NaOH to produce cellulose and lignin, which remains connected to hemicellulose [6]. Therefore, the amount of cellulose in the fiber increases while hemicellulose and lignin decrease, because they dissolve in water. Based on the findings of this test, we may deduce that modification of the fiber surface using alkalization is the right method for improving the characteristics of bagasse fibers, which can increase the cellulose content and reduce the hemicellulose and lignin content in the fiber.

FTIR Analysis of Bagasse Fiber

FTIR spectrum analysis was conducted to determine the effect of alkalization on functional groups in bagasse fibers. The bagasse fibers used were before and after alkalization with 7% (w/vol) NaOH. Meanwhile, the functional groups observed in this study include hydroxyl functional groups (-OH) in cellulose, hemicellulose, and lignin, shown at wave number 3340.64 cm^{-1}, carboxyl functional group (C=O), which indicates the presence of hemicellulose, shown at wave number 1725.90 cm^{-1}, and carbon functional group (C=C), which indicates the presence of lignin, shown at wave number 1611.05 cm^{-1}[24], as well as alkane functional groups (cellulose, hemicellulose, and lignin) shown at wave number 1243.81 cm^{-1} [25]. The results of FTIR characterization in this study, are shown in **Figure 4**.

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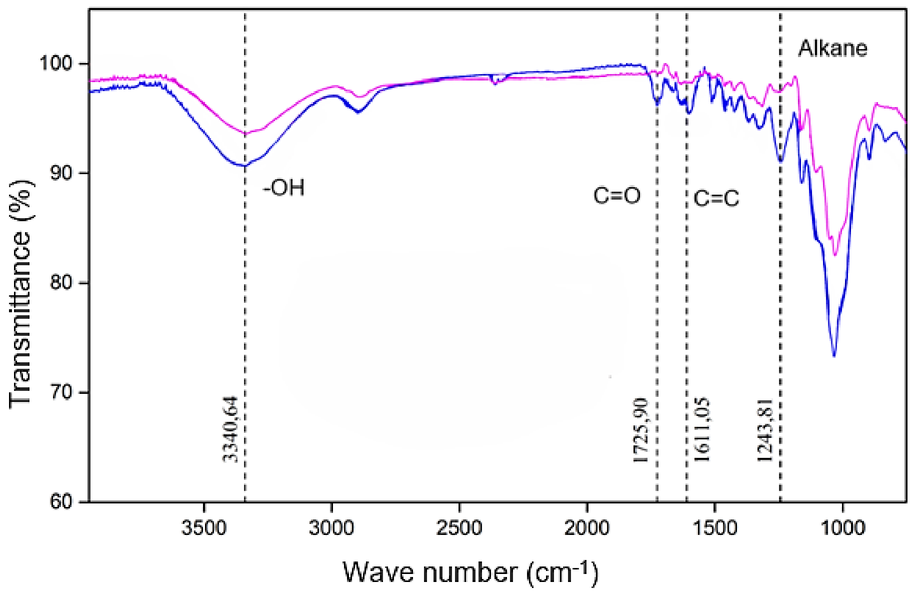

Figure 4. The FTIR spectrum of Bagasse fibers after alkaline treatment with 0% NaOH (blue color) and 7% NaOH (pink color).

From the results of the FTIR spectrum analysis in **Figure 4**, it is known that the number of hydroxyl functional groups (-OH) of bagasse fiber decreased after alkalization treatment, as indicated by an increase in the wave spectrum peak at wave number 3340.64 cm^{-1}. This means that the hydrophilic nature of bagasse fiber decreased after alkalization. Then, the amount of hemicellulose in bagasse fiber decreased after alkalization treatment. The decrease in the amount of hemicellulose is indicated by the increase in the peak of the wave spectrum of the carboxyl (C=O) functional group at wave number 1725.90 cm^{-1} and the alkane functional group at wave number 1243.81 cm^{-1}. Like hemicellulose, the amount of lignin in the fiber also decreased after alkalization treatment, shown at wave number 1611.05 cm^{-1} by the carbon (C=C) functional group [24]. From the results obtained, it can be concluded that the surface modification of bagasse fiber using the alkalization method can reduce the hydrophilic nature of the fiber, the hemicellulose component, and lignin in bagasse fiber. This follows the results of research by Bartos [27], where NaOH alkalization treatment of bagasse fiber with increasing NaOH concentration up to 40% can reduce the amount of hemicellulose with the C = O functional group at the peak of 1730 cm^{-1} and lignin with the C = C functional group at the peak of 1514 cm^{-1}. The decrease in the amount of hemicellulose and lignin in the fiber after alkalization is caused by structural damage to the fiber, which affects fiber's composition [27].

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Conclusions

This study successfully demonstrated the alkalization process's effectiveness in improving sugarcane bagasse fiber’s characteristics as potential reinforcement materials for composites. The key findings are as follows:

1. The alkalization process using 7% (w/vol) NaOH significantly reduced the diameter of bagasse fibers from 3.7 µm to 0.35 µm, indicating the removal of non-cellulosic components like lignin and hemicellulose.
2. The rougher and fibrillated surface of the treated fibers increased their specific surface area, enhancing interfacial bonding potential with polymer matrices.
3. Alkalization treatment resulted in a significant increase in cellulose content (79.02%) and a marked decrease in hemicellulose (0.82%) and lignin (10.15%) compared to untreated fibers. This indicates better suitability for high-performance composite materials.
4. The reduction in hydrophilic hydroxyl (-OH) groups and the decomposition of hemicellulose and lignin were confirmed by FTIR spectroscopy. The decreased hydrophilicity enhances the compatibility of fibers with hydrophobic polymer matrices.

This research introduced a more sustainable approach to processing sugarcane bagasse fibers by utilizing a lower NaOH concentration (7% w/v) compared to previous studies and incorporating acid neutralization for better chemical balance. Additionally, the pre-treatment process ensured the removal of cork residues, which was not explored in earlier works.

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